

FEU11 – SOP for the Modified Griess Test for Nitrite Residues

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1. Scope

- 1.1. This procedure is designed to provide specific technique for detecting and preserving patterns of nitrite residues around a suspected bullet hole(s) as a basis for estimating muzzle-to-target distances. Such patterns may be on evidence items to include but not limited to clothing, furniture, bedding, and wallboard.

2. Background

- 2.1. To establish the practices for documenting the examination of firearm evidence to conform to the requirements of the Department of Forensic Sciences (DFS) Forensic Science Laboratory (FSL) *Quality Assurance Manual*, the accreditation standards under ISO/IEC 17025:2005, and any supplemental standards.

3. Safety

- 3.1. For proper handling of firearm see the *FEU01 - SOP for the Safe Handling of Firearms*.
- 3.2. Ensure the proper ventilation is provided during both the preparation of the reagents and the procedure itself. When handling clothing that is potentially contaminated with biological hazards or preparing reagents for chemical tests, protective latex or vinyl gloves, safety glasses, and laboratory coat will be worn at all times.

4. Materials Required

4.1. Table of Materials Required

Scissors	Beaker
Cheesecloth	Latex/Nitrile gloves
Graduated Cylinder	Tweezers
Brown wrapping paper	Flat iron
Processing tray	Spatula
Photographic paper	Exhaust hood
Wire brush	Polyethelene bottles
Filter Paper	Sodium Nitrate (Reagent grade or better)
Sulfanilic acid (reagent grade or better);	Glacial acetic Acid (reagent grade or better)
Alpha-naphthol (reagent grade or better);	Methanol (reagent grade or better);
Distilled Water	Resealable bags (i.e. ziplock type)
Cotton swabs	Lab Coat

5. Standards and Controls

- 5.1. Positive and negative controls are prepared for the Modified Griess test to detect nitrites. If a chemical reagent must be prepared before an examination, the following must be recorded on the FEU Chemical Preparation Log (Appendix A): date prepared, examiner/technician's initials, chemical prepared, and the amount prepared.

6. Calibration

- 6.1. Not applicable

7. Procedures

- 7.1. Preparation of Nitrite Test Swabs

- 7.1.1. Prepare a solution of 0.6 grams of sodium nitrite in 100 ml of distilled water in a beaker.
- 7.1.2. Divide the package of cotton swabs into equal amounts. Soak half of the cotton-tipped ends in the nitrite solution until saturated. Dispose of any remaining solution as outlined in the Hazardous Waste Disposal section in the FEU Chemical Preparation Book.
- 7.1.3. Set the swabs aside to dry in the fume hood. Once dry, store in a labeled resealable bag. These will serve as the positive control. There is no known limit to the shelf life of these swabs.
- 7.1.4. Retain the non-treated swabs in a re sealable bag. These swabs are used for the negative control.

7.2. Preparation of Reagents and Test Media

- 7.2.1. The following instructions apply to the preparation of the reagents and test media for use in the Modified Griess Test for nitrite residues. The desensitized photo paper mentioned below is simply photographic print paper which no longer bears light-sensitive silver salts in its surface emulsion. Proper procedures for the disposal of the chemicals used are outlined in the to the Hazardous Waste Disposal section in the FEU Chemical Preparation Book.

7.3. Processing of Previously Desensitized Photographic Paper

- 7.3.1. Prepare a solution of 0.5 grams of sulfanilic acid in 100ml of distilled water in a beaker.
- 7.3.2. Prepare a solution of 0.28 grams of alpha-naphthol in 100ml of methanol in a beaker.
- 7.3.3. Combine the solution from step 2 into the solution from step 1 in a beaker.
- 7.3.4. Pour the combined solution into a non-reactive photo processing tray and briefly dip pre-cut sheets of the photographic paper into the tray. Submerge the sheets completely. Remove them and let air dry. Note: as a substitute for photographic paper, ordinary laboratory filter paper may be processed in the same manner. (See the suggested method in the specific test procedure which follows.)
- 7.3.5. Set the sheets aside to dry. Dispose of any remaining solution as outlined in the Hazardous Waste Disposal section in the FEU Chemical Preparation Book.

7.4. Preparation of a 15% Acetic Acid Solution

- 7.4.1. Combine 150ml of glacial acetic acid with 850ml of distilled water. Gently pour the acid into the water to preclude the potential spattering of undiluted acid.
- 7.4.2. Store and label the solution in a properly sealed container with the identity of the reagent, date of preparation or lot number and the preparer's initials.

7.5. Enter information into the FEU chemical preparation log.

7.6. Procedure for a Standard Modified Griess Test

- 7.6.1. To ensure desensitized and chemically treated photographic paper or filter paper is functioning properly; test the four corners of the emulsion-coated side with a positive control. This is accomplished by saturating a nitrite test swab (positive control) in a small amount of 15% acetic acid solution and dabbing the four corners of the paper. An orange color should appear at each corner, confirming sensitivity. The results will be recorded in the examination notes.
- 7.6.2. For a negative control, repeat the above step, but use clean, non-nitrite treated acetic acid-saturated test swabs. Ensure that this test follows the positive control test, and that there is sufficient physical separation between test marks to preclude bleeding from one mark to the other. The results will be recorded in the examination notes.
- 7.6.3. Place the evidence/questioned item or known-distance test questioned side down on the emulsion-coated side of the treated photographic paper. Index such things, i.e. seams, button holes, rips, pockets, suspected bullet holes, tears, cuts, etc...for possible future reference by marking with a pencil. DO NOT USE INK at this point because it may transfer back onto the tested item.
- 7.6.4. Soak a piece of cheesecloth in the 15% acetic acid solution (in a large beaker) and wring out to dispose of excess liquid. Place the cheesecloth on the evidence/questioned item or known-distance test as the third layer of the "sandwich". Press the "sandwich" with a hot iron. On many irons, the setting for "cotton" is appropriate.
- 7.6.5. Discard the cheesecloth into the biohazard container. Separate the evidence/questioned item or known-distance test-firing from the photographic paper.

- 7.6.6. When dry, the photographic paper should be marked appropriately. (Note: photographic paper is considered secondary evidence and will be marked with the FEU #, Item #, hole # being processed, date and initial of the examiner. Retain any photographic paper showing positive results as part of the raw data for inclusion in your notes. Test media relating to negative results need NOT be retained.

7.7. The Modified Griess Test Using Treated Filtered Paper

- 7.7.1. Treat the filter paper in the same solutions used for treating the photographic paper. Allow it to dry. (See Processing of Previously Desensitized Paper).

- 7.7.2. Place the filter paper on the questioned surface. Test for nitrite sensitivity using positive and negative controls.

- 7.7.3. Process the filter paper using ONE of the following methods in the fume hood:

- 7.7.4. Saturate a piece of cheesecloth in the 15% acetic acid solution and wring out. Place the cheesecloth over the filter paper and apply a hot iron.

OR

Spray the filter paper with 15% acetic solution until very damp. Cover with two or three additional layers of the filter paper and iron until dry.

- 7.7.5. Separate the test media and check results.

- 7.7.6. When dry, mark and preserve results for retention as in the above procedure.

7.8. Procedure for a Reverse Modified Griess Test for Thick or Otherwise Non-Porous Materials Through Which the Acetic Acid Solution "Steam" Will Not Penetrate

- 7.8.1. Tape a piece of filter paper or other appropriate nitrate-free substitute to the back of the piece of desensitized and treated photographic paper. Test as in the above procedures using nitrite test swabs.

- 7.8.2. Place the photographic paper emulsion side down on the questioned surface and use a pencil to index seams, buttons, suspected bullet holes, pockets, rips, tears, cuts, etc., for possible future courtroom reference.

- 7.8.3. Wipe the emulsion-coated side of the photographic paper with a piece of cheesecloth saturated with a 15 % acetic acid solution. Apply the solution

to the entire surface, but lightly. Too much will cause indistinct or hazy results due to pigment migration.

- 7.8.4. Immediately place the photographic paper emulsion side down on the questioned surface. Apply a hot iron to the back of the photographic paper until dry. Note that the back was previously covered by filter paper or an appropriate substitute; otherwise the paper may stick to the iron.
- 7.8.5. Separate the photographic paper and the questioned item. Any orange indications on the photographic paper are the result of the presence of nitrite residues.
- 7.8.6. When dry, mark and preserve any positive results for retention as in the previously described normal Modified Griess Test.

8. Sampling

- 8.1. Not applicable

9. Calculations

- 9.1. Not applicable

10. Uncertainty of Measurement

- 10.1. When quantitative results are obtained, and the significance of the value may impact the report, the uncertainty of measurement must be determined. The method used to determine the estimation of uncertainty can be found in the *FSL Quality Assurance Manual – Estimation of Uncertainty of Measurement (Section 5.4.6)*.

11. Limitations

- 11.1. This test is not appropriate for the detection of purely nitrate compounds, such as unburned smokeless powder. A practical reality, however, is that unburned powder particles (nitrates) are commonly coated with burned powder residues (nitrites) and positive reactions take place. The Modified Griess Test yields results for nitrite residues regardless of whether these are in fact gunshot residues.

12. Documentation

- 12.1. FEU Chemical Preparation Log
- 12.2. Gunshot Residue Work sheet(s)
- 12.3. Photos (When Applicable)
- 12.4. FEU Report of Results

13. References

- 13.1. Federal Bureau of Investigation Gunpowder and Gunshot Residue Manual (current revision)
- 13.2. FEU Chemical Preparation Book (Current Version)
- 13.3. *DFS Health and Safety Manual* (Current Version)
- 13.4. *Forensic Science Laboratory Quality Assurance Manual* (Current Version)
- 13.5. *FSL Departmental Operations Manuals* (Current Versions)
- 13.6. *FSL Laboratory Operations Manuals* (Current Versions)
- 13.7. *FEU01 - SOP for Safe Handling of Firearms* (Current Version)